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IS 12981 (1991): Common Salt - Iron Fortified [FAD 8: Food Additives]

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Indian Standard

COMMON SALT — IRON FORTIFIED — SPECIFICATION

UDC 664·41

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 1991 **Price Group 2**

Acids, Alkalies and Halides Sectional Committee, CHD 02

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

Iron deficiency anaemia is a major nutritional problem in our country. The vulnerable groups most affected are women of reproductive age, pregnant women and pre-school children.

The major cause of anaemia in our country is iron deficiency due to inadequate intake of dietary iron and/or its poor absorption from predominantly cereal based diets comsumed by our population. One measure to prevent iron deficiency anaemia is to increase iron intake in the diet through fortification of a universally consumed dietary item with iron. In India, edible common salt has been identified as a suitable material for fortification by iron as it is comsumed in fairly uniform quantity by every section of the population.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this regard.

Indian Standard

COMMON SALT — IRON FORTIFIED — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for iron fortified common salt.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjunct to this standard:

IS No.	*Title*
253 : 1985	Specification for edible common salt (*third revision*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)
2711 : 1979	Direct reading *p*H meters (*second revision*)
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

3.1 Description

The material shall be crystalline solid, white or pale pink or light grey in colour, free from visible contamination with clay, grit, other extraneous adulterants and impurities.

3.2 Moisture

The material as received shall contain not more than 5·0 percent by mass of moisture when tested in accordance with the method prescribed in **A-2** of IS 253 : 1985.

3.3 Particle Size

95 percent of the material shall pass through 4·00 mm IS Sieve.

3.4 The material dried in accordance with the method prescribed in **A-2** of IS 253 : 1985 shall also comply with the requirements given in Table 1 when tested according to the relevant methods prescribed in IS 253 : 1985 and Annex A.

4 PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in HDPE bags or any other suitable packages, as agreed to between the purchaser and the supplier.

4.1.2 For bulk packings, the material may be packed in 40, 50, 75 and 100 kg packages.

4.1.3 For small packings, the material may be packed in 50 g, 100 g, 200 g, 500 g, 1 kg, 2 kg and 5 kg suitable packages.

Table 1 Requirements for Common Salt — Iron Fortified

(*Clause* 3.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Clause No. in Annex A	Method of Test, Ref to Clause No. in IS 253 : 1985
i)	Water insoluble matter, percent by mass, *Max*	1·0	—	A-4
ii)	Chloride content (as NaCl), percent by mass, *Min*	96·5	—	A-5
iii)	Matter insoluble in dilute hydrochloric acid, percent by mass, *Max*	0·3	—	A-6
iv)	Matter soluble in water, other than sodium chloride, percent by mass, *Max*	2·5	—	A-7
v)	Magnesium (as Mg), water soluble, percent by mass, *Max*	0·1	—	A-8
vi)	Sulphates (as SO_4), percent by mass, *Max*	1·1	—	A-9
vii)	Iron (as Fe), percent by mass	0·085 to 0·110	A-2	—
viii)	*p*H of 5 percent aqueous solution	2·0 to 3·5	A-3	—
ix)	Phosphates (as PO_4), percent by mass	0·15 to 0·20	A-4	—

4.2 Marking

The packages shall be securely closed and marked as follows:

a) Name 'IRON FORTIFIED COMMON SALT',

b) Indication of the source of manufacture,

c) Lot and/or batch No. and date of manufacture,

d) Net mass of the material, and

e) CAUTION — STORE IN A COOL AND DRY PLACE.

5 SAMPLING AND CRITERIA FOR CONFORMITY

5.1 Representative samples of the material shall be drawn and their criteria for conformity shall be determined in accordance with the methods prescribed in Annex B.

ANNEX A

(*Clause* 3.4 and *Table* 1)

METHODS OF TEST FOR IRON FORTIFIED COMMON SALT

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF IRON

A-2.0 Outline of the Method

Iron is determined colorimetrically by measuring the transmittance of reddish-violet colour produced by ferrous and ferric ion with thioglycollic acid at *p*H 10.

A-2.1 Apparatus

A-2.1.1 *Photoelectric Absorptiometer*

A-2.2 Reagents

A-2.2.1 *Dilute Hydrochloric Acid*

Approximately 5 N.

A-2.2.2 *Sodium Citrate Solution 1 M*

29·4 g per 100 ml, or alternatively ammonium citrate solution (2 M) 43·0 g per 100 ml may be used.

A-2.2.3 *Ammonium Hydroxide*

Approximately 16 N.

A-2.2.4 *Thioglycollate Reagent*

Add 20 ml of ammonium hydroxide to 30 ml of water, and then add a mixture of 10 ml of thioglycollic acid and 40 ml of water.

A-2.2.5 *Standard Iron Solution*

Dissolve 0·491 1 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2SO_4 \cdot 6H_2O$] in water, and add 10 ml of dilute sulphuric acid (10 percent *v/v*). Dilute to exactly 100 ml in a volumetric flask. Take 25 ml of this solution into a 250 ml volumetric flask and dilute to the mark with water. One millilitre of this solution contains 0·07 mg of iron (as Fe).

A-2.3 Procedure

A-2.3.1 Weigh accurately 1 g of the material and transfer to a 150-ml beaker. Add 30 ml of water and 5 ml of dilute hydrochloric acid. Introduce a glass rod, cover with a watch glass and boil gently for 15 to 20 minutes. Cool and transfer to a 100-ml volumetric flask and dilute to about 70 ml. Place 5 ml of dilute hydrochloric acid and 65 ml of water in a second flask as blank.

A-2.3.2 To each flask, add 4 ml of sodium citrate solution (or 2 ml of ammonium citrate solution), 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide solution, swirling after each addition. Dilute to the mark and mix well.

A-2.3.3 Balance the absorptiometer at blank using green filter No. 4 and 4-cm cells. (Use 1-cm cell if the colour is too strong). Find the reading with the sample solution. Read off milligram iron from the standard calibration graph (*see* **A-2.3.4**).

A-2.3.4 *Standard Calibration Graph*

Pipette out known volumes of standard iron solution into each of 100-ml volumetric flask containing 5 ml of dilute hydrochloric acid. Proceed as in **A-2.3.2**. Suitable aliquots are 0, 0·1, 1·5, 2·0, 2·5 and 3·0 ml. Take the readings. Plot graph with milligram iron as ordinate and absorptiometer readings as abscissa. Draw a line through the points and extend through the origin. Label the graph with particulars of filters and cells used.

A-2.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{M_1}{10 \times M}$$

where

M_1 = mass, in mg, of iron (as Fe) in 100 ml of the sample solution; and

M = mass, in g, of the material taken for the test.

A-3 DETERMINATION OF *p*H

A-3.1 Apparatus

A-3.1.1 *p*H *Meter*

Use a standard laboratory *p*H meter (*see* IS 2711 : 1979.

A-3.2 Procedure

Dissolve 20 g of the sample in 100 ml water. Measure the *p*H of the solution using a standard *p*H meter. Repeat the test and record the mean *p*H value.

A-4 DETERMINATION OF PHOSPHATE CONTENT

A-4.0 Outline of the Method

Phosphates develop the molybdenum blue colour and the intensity is measured by spectrophotometer.

A-4.1 Apparatus

A-4.1.1 *Spectrophotometer,* with a 650-nm filter.

A-4.2 Reagents

A-4.2.1 *Standard Phosphate Solution*

Dissolve 0·046 2 g of pure and dry potassium dihydrogen phosphate (KH_2PO_4) in water and add 10 ml of ammonium acetate/acetic acid buffer solution of *p*H 5. Dilute the mixture with water and make up the volume to 1 000 ml.

A-4.2.2 *Reducing Agent Solution* — 0·15 percent (*m/v*).

Dissolve 1·5 g of pure and recrystallized 1-amino-2 naphthol-4-sulphonic acid and 7 g of sodium sulphite (anhydrous) in 75 ml of water. Mix this solution with a solution containing 90 g of sodium bisulphite in 700 ml water. Dilute the final solution to 1 000 ml with water and store in a dark glass bottle.

NOTE — Avoid direct light during the preparation of reducing agent solution.

A-4.2.3 *Sulphuric Acid,* 8 *N*

A-4.2.4 *Ammonium Molybdate Solution*

Dissolve 100 g of ammonium molybdate in sufficient quantity of water and dilute to 1 000 ml with water.

NOTE — Fresh solution should be prepared if insoluble matters are formed due to ageing.

A-4.3 Procedure

A-4.3.1 Weight about 1 g of the sample and dissolve in sufficient quantity of water. Transfer this solution to a 100 ml measuring flask and dilute with water up to the mark. Take 5 ml of this solution in a 100 ml measuring flask and add 3 ml of 8 N sulphuric acid, 1 ml of 10 percent (*m/v*) of ammonium molybdate solution and 2 ml of reducing agent (*see* **A-4.2.4**). Dilute the solution to the mark with water and measure the absorbance at 650 nm.

A-4.3.2 *Preparation of Calibration Curve*

Transfer 1·5, 3, 4·5, 6·0 and 7·5 ml of standard phosphate solution in different 100 ml measuring flask. Add 3 ml of 8 N sulphuric acid, 1 ml of 10 percent (*m/v*) ammonium molybdate solution and 2 ml of the reducing agent solution (*see* **A-4.2.4**) to each of the measuring flasks and dilute to 100 ml with water. Measure the absorbance of each solution at 650 nm after 30 minutes. Prepare a calibration curve by plotting absorbance against concentration of standard phosphate solution and determine the concentration of unknown sample (*see* **A-4.3.1**) from the calibration curve by reading absorbance.

A-4.4 Calculation

$$\text{Phosphate (as } PO_4 \text{), percent by mass} = \frac{M \times 100}{M_1}$$

where

M = phosphate content, in g, in 100 ml of the sample solution; and

M_1 = mass, in g, of the material taken for the test.

ANNEX B

(*Clause* 5.1)

SAMPLING OF IRON FORTIFIED COMMON SALT

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken out in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The sample shall be placed in suitable, clean, dry and air-tight glass containers.

B-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and year of manufacture.

B-2 SCALE OF SAMPLING

B-2.1 *Lot*

All the packages in a single consignment of iron fortified common salt drawn from a single batch of manufacture shall constitute a lot. If the consignment consists of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

B-2.1.1 The number of packages (*n*) to be selected shall depend upon the size of the lot (*N*) and shall be in accordance with Table 2.

B-2.1.2 These packages shall be selected at random from the lot, and to ensure randomness of selection, a random number table (*see* IS 4905 : 1968) may be used.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 Packages

B-3.1.1 From each of the packages selected in accordance with **B-2.1.2**, a suitable portion of

Table 2 Number of Packages to Be Selected for Sampling

(*Clause* B-2.1.1)

Lot Size n	No. of Packages to be Selected N
4 to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

NOTE — When the size of the lot is 3 or less, the number of packages to be selected and the criterion for judging conformity for the lot to the specification shall be as agreed to between the purchaser and the supplier.

the material (about 200 g) shall be drawn with the help of a suitable sampling instrument.

B-3.1.2 Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 600 g. The composite samples shall be divided into three equal parts, namely, one for the purchaser, the other for the supplier and the third to be used as a referee sample.

B 3.1.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual sample representing the container samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.2 All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.3 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of moisture content, chloride content (as NaCl) and level of iron shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all other characteristics given under **3** shall be performed on the composite sample (*see* **B-3.1.2**).

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

From the test results the average (X) and the range (R) or (R) shall be computed for each of the characteristics mentioned in **B-4.1**. Range (R) is defined as the difference between the maximum and the minimum values of the individual test results. Range ($\bar{R}$) is the average of two ranges calculated on each of the two sets of five consecutive test results when the total number of test results on a characteristic is 10. The criteria for conformity of the lot in respect of each of these characteristics is that ($x - 0{\cdot}6\,R$) or ($x - 0{\cdot}6\,\bar{R}$) should be greater than or equal to the corresponding minimum requirements specified under **3**.

B-5.2 For Composite Samples

The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample, if the test results satisfy the corresponding requirements given under **3**.

B-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **B-5.1** and **B-5.2**.

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Doc : No. CHD 02 (9461)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Swatantra Bharat Press, Delhi, India